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³¹P and ¹³C NMR Investigations of a Tert Butylcalix[4]Arene-Diphosphate

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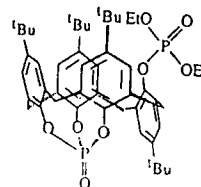
^{31}P AND ^{13}C NMR INVESTIGATIONS OF A TERT.BUTYLCALIX[4]ARENE-DIPHOSPHATE

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Abstract. The lanthanide induced ^{31}P and ^{13}C shifts for calix[4]arenediphosphate **1** are studied. From these investigations an exact assignment of all the 48 C atoms was possible.

The aim of our investigations was to perform an exact assignment of all the 48 C atoms in the tert.butylcalix[4]arenediphosphate **1**. In the ^{31}P NMR spectrum of **1** we found two signals in the typical region. It is remarkable that the shift behaviour of the cyclic and the acyclic phosphate group is very different. By addition of $\text{Eu}(\text{FOD})_3$ the acyclic P atom shifts very strongly towards high-field but the chemical shift of the cyclic P atom is constant². This behaviour is due to a steric effect of the aromatic ring with the tert.butyl substituent in the near of the cyclic P=O group. In the ^{13}C spectrum of **1** we found 34 signals, and after addition of $\text{Eu}(\text{FOD})_3$, 36 signals. By adding the shift reagent the chemical shifts of all C atoms change but not the coupling constants. The shift effect is stronger in the near of the acyclic phosphorus and far from the acyclic phosphorus this effect is weaker.



partial cone form

1

An exact assignment of all the 48 C atoms can be deduced from a combination of the chemical shift values of the C atoms before and after adding $\text{Eu}(\text{FOD})_3$, the magnitude of the coupling constants, and the peak intensities.

REFERENCES

1. J. GLOEDE, B. COSTISELLA, M. RAMM and R. BIENERT, *Phosphorus, Sulfur, and Silicon*, **84**, 217 (1993).
2. B. COSTISELLA and J. GLOEDE, *Phosphorus, Sulfur, and Silicon*, **89**, 39 (1994).